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SOLID PROPELLANT AGING STUDIES

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CLEVELAND S. WATERMAN

AUGUST 1976

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FOREWORD

This report was submitted by the Propellant Formulation and Ingredients Section (MKPA) of the Air Force Rocket Propulsion Laboratory, Edwards AFB, California 93523 under Job Order 305908NX.

This report has been reviewed by the Information Office (DOZ) and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations.

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Since 1972 AFRPL has been building up an In-House capability for aging solid propellants. By November of 1974 the facilities had expanded to the point where full scale aging of propellants could be performed. A three-year plan for the accelerated aging of 17 different solid propellant formulations was prepared. The aging conditions include elevated temperatures and humidity. Data has been compiled but no analysis of this data has as yet been accomplished.

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PREFACE

This technical report summarizes the work done on the In-House Propellant Aging Program at the AFRPL between 1 November 1974 and 30 June 1976. No previous technical reports have been published on this program.

The authors wish to acknowledge those people who proved invaluable to the development of an aging capability at the AFRPL. MSgt. Louis Franks, TSgt. Rex Thompson and Mr. Kelley Palmer were responsible for planning and overseeing the necessary construction of the facility. They also had the difficult task of day-by-day maintenance of the environmental chambers and cutting the samples. The actual aging of the propellants is only a part of the task. An important part is sample testing, and Mr. Tom Chew has done an excellent job at this.

SUMMARY AND CONCLUSIONS

Since 1972, the AFRPL has been building up an In-House capability for aging solid propellants. This initial work involved the selection of a site for the activities and the procurements of equipment that would make accelerated aging of propellant possible. This equipment consisted mostly of environmental chambers that could store propellant for long periods of time at constant humidity and temperature. By November of 1974, the facilities had expanded to a point where full scale aging of propellants could occur.

Once the facilities were in place and operating, a standard procedure for utilizing the capabilities had to be formulated. This procedure includes the timely insertion of propellant into the environmental chambers along with the subsequent extraction and testing. Coordination of all facilities and personnel had to be effected. This technical report summarizes how this task was accomplished. Now that the capability and procedures have been established, future reports will contain the actual aging data.

INTRODUCTION

During the early 1970's, hydroxy terminated polybutadiene (HTPB) propellants graduated from laboratory curiosities to leading contenders for future solid rocket motors. The two primary composite propellant systems with which HTPB is competing are carboxy terminated polybutadiene (CTPB) and polybutadiene - acrylic acid - acrylonitrile (PBAN). The latter two systems have been in use since the 1950's and are well characterized with a massive amount of aging data available (reference #1). No matter how favorably HTPB may compare in regards to cost, processing, and initial mechanical properties, the available aging data and experience with PBAN and CTPB propellants are important criteria to systems personnel and prime contractors. In order to lend credence to the AFRPL's contention that HTPB is ready for applications, aging capability must be established.

The years of experience necessary for establishing that capability is lacking. However, accelerated aging has indicated (references #2 and #3) the superior aging characteristics of HTPB propellants. Later in this report, the danger of overinterpretation or misinterpretation of accelerated aging data will be pointed out.

It is the objective of this report to review the efforts which have led to the development of the capability to perform an aging study at the AFRPL and to review the types of propellants currently being aged. The reason for performing aging studies at the AFRPL is to eliminate comparison problems among different facilities by having a central facility to age and test propellants under identical conditions.

The equipment and experimental procedures will be discussed in detail in a subsequent section of this report. In essence, the AFRPL has developed the capability to age propellants under a variety of conditions. In addition to bunkers and storage rooms for long-term, ambient aging, chambers are available for storing under a variety of temperature/humidity conditions.

It is an understood fact that many problems exist in aging propellants in cartons. Primarily, the correlation of carton sample data to dissected motor sample data has been relatively poor (reference #4). This is particularly true for

large motors where the exothermic cure reaction can produce up to 9°F higher temperatures in the center of the grain during cure. The temperature differential results in different propellant properties. One way around this problem is to determine the temperature gradient across the grain during cure and then to cure cartons at the required temperatures to correspond to certain sections of the motor. An additional problem with carton aging is that flow lines are introduced during casting and the physical properties (as well as burn rate) will vary with these lines. The flow (and thus properties) can be different in cartons and motors, and in different parts of a motor.

Reproducibility of test methods is another problem associated with interpretation of aging data. This includes such factors as: sample containers (e.g., unlined, foil-lined, lined with insulation/liner as per motor); means of preparing JANNAF specimen (e.g., stamp, mill, Class A, B, or C); time from cutting to testing and; control of humidity and temperature in the Instron Chamber. These reproducibility problems sometimes result in data so erratic that aging trends are obscured by data scatter. The problems are compounded to an insurmountable degree if one tries to make facility-to-facility comparison of aging trends. This latter fact is a strong justification for having one central facility for aging and testing propellants under identical conditions.

Accelerated aging methods and data interpretation have always been questionable. The attempts to perform aging studies at elevated temperatures and project age life at a lower temperature ignores the probability of having reactions which occur at one temperature but are of no consequence at a lower temperature. A major step towards resolving this question has been the work of Layton (reference #2 and #5) which entails gathering chemical aging data along with mechanical property aging data at different temperatures to assure linear changes in both. If, for instance, a linear plot is obtained for gel content vs. log time at temperatures up to 150°F and linear plots are obtained for some property (e.g., stress) over the same temperature range, it may be assumed that the same aging mechanism is applicable over that range. Therefore, a simple Arrhenius relationship may be used to derive a rate constant from $dp/dt = k/t$ and predict property changes (dp) over a time period (dt).

A practical problem encountered in predicting age life (even assuming a knowledge of temperature effects) is the uncertainty of cyclic effects. An ICBM

may see a relatively constant temperature whereas a tactical missile is exposed to a varied temperature history. Davis and Nelson (reference #6) have shown for an HTPB propellant that total time at an elevated temperature is the important factor and the same degree of damage occurs regardless of whether the exposure for that time period is continuous or intermittent.

One final environmental aging condition to be considered is humidity. Most motors have weather seals. However, some are open to moisture and others suffer punctures of weather seals. It would be valuable to know the effect of moisture and one objective of this In-House effort is to determine that effect.

All of the propellants currently at the AFRPL were produced on various contracts with the industry. A large number are under investigation because they cover a wide burn rate range (0.22 to 2.1 in/sec at 1000 psia) and act as baselines for many different systems. They also serve as means of comparing various ingredients. For instance, the UTP propellants will give comparative data on two different Pro-Techs and a standard antioxidant with all other factors being identical. The ANB formulations look at the long term metal cure catalysts effects along with a different antioxidant blend. The TP-H1135 is an example of a propellant with no plasticizer or metal additives. TP-H8213 will give data on DDI as the curative. TP-H8219 and 8220 will give comparative data on two means of getting to a high rate. One way has UFAP and the other transparent iron oxide.

EXPERIMENTAL DETAILS

At present, the propellant aging work at Test Area 1-21 breaks down into two distinct phases. First is a two-year long period during which the propellant is stored under a variety of environmental conditions and is sampled and tested at predetermined intervals. Second is an indefinite period of storage during which no testing is planned. The propellant is available for testing should any aspect of that propellant be of possible future interest. For example, the importance of propellants containing ferric fluoride was not known at the time of their receipt at the AFRPL. However, the effect of ferric fluoride on the aging of reduced smoke Maverick and SRAM propellants became of concern, and therefore the ferric fluoride containing propellant was tested one year after being placed in the AFRPL inventory. The second phase is simple. All that is needed is a shelf on which the propellant can be stored under ambient conditions of temperature and humidity. Phase I requires a great deal more planning, more facilities, and more data handling. The primary thrust of this report will be to show how Phase I was constructed and how it can be modified to permit the easy insertion of new propellants into the two year formal period of aging.

Since the two years is not considered long in the lifetime of a propellant, an environment that would accelerate the aging process of the propellant is required. Thus, the six environmental chambers at Test Area 1-21 are the heart of the In-House Propellant Aging Program. Figure 1 gives a technical description of the chambers being used. Prior to 1972, environmental control capability at the AFRPL was very limited. Humidity control was obtained by placing various salt solutions in ovens. Varying humidity was obtained by varying the solution. The chambers for temperature control were in the open with only a sun shade over the top and dirt bunkers on three sides. Under these conditions it was difficult to maintain desired environments. It was also difficult to maintain the chambers in operable condition due to exposure to the elements. Thus, it was decided that chambers capable of maintaining both temperature and humidity and stationed in an enclosed area would be the best conditions for an aging program. This is the arrangement being employed at Test Area 1-21.

The proper operation and maintenance of the ovens is critical to the entire program. Years of aging could be wiped out overnight if the ovens malfunctioned.

Model No.	Tenney TH65	Missimer FTH8X250
Temperature Range	$0^{o}F$ - $200^{o}F$	Ambient - $250^{o}F$
Temperature Tolerance	$\pm 1^{o}F$	$\pm 3^{o}F$
Relative Humidity Range	20% RH - 95% RH	20 - 80% RH
Relative Humidity Tolerance	±5% RH	±5% RH
Heating	Electric Air Heater	Electric Air Heaters
Dehumidification	Refrigeration	Air Passage Over Cool Water
Humidification	Immersion Heater	Immersion Heater

Figure 1. Environmental Chamber Specifications

For example, the temperature controller could go astray and literally bake the propellant. Just as bad, the humidity controller could fail and drown the propellant. In the early years of the program both of these fatal malfunctions occurred. To prevent their future occurrence, redundant high temperature cutoffs were installed on both the wet and dry bulb temperature controls. If either of the set points on these redundant controllers is exceeded, the entire oven is shut down. For example, a very high wet bulb temperature indicates that the relative humidity is high since the wet air can no longer cool off the wick in the wet bulb temperature sensor. To completely avoid any break in the test schedule, one of the six ovens is held on a standby basis to take over the duties of any one of the other five ovens that malfunctions and shuts down. When such a malfunction occurs, the party responsible for effecting repairs is Component Processing, also known as Chalco, Inc. The duties of this organization are described more fully in Appendix I.

The actual use of the ovens is relatively simple. The bulk samples of propellant are cut into blocks with the rough dimensions 5 in. x 4 in. x 3 in. for a short test series and 5 in. x 8 in. x 3 in. for a full test series. The series will be described subsequently. A sufficient number of these blocks for carrying out the two years of formal aging and testing are placed into the ovens. The blocks are wrapped on all sides except one so that any gradient effects can be measured. The blocks are placed in the ovens so that the exposed surface faces the door of the chamber. If the exposed surface of the block were to face upwards, any collection of water on the upper surface could pool. This is, of course, undesirable. Care is also taken to avoid stacking the blocks on top of each other so that the physical stress on all blocks is the same. Propellant is also stored under ambient conditions for both Phase I and Phase II. Gradient effects are considered negligible in ambient storage. No special wrapping precautions are taken. The propellant is stored in various bulk sizes and various types of wrapping. For the most part, however, the propellant in ambient storage is wrapped much like that which is placed in the ovens.

After storage comes testing. The mechanical properties testing is, at present, limited to uniaxial Instron testing. Thus, the only treatment of the bulk propellant necessary is the cutting, milling, and stamping of the dogbones used in Instron testing. Throughout the program, the tolerance of the dimensions of the dogbones conform to the specifications laid down for a JANNAF Class C dogbone. Figure 2 gives these dimensions. The rough cutting is accomplished using a Blue Chip

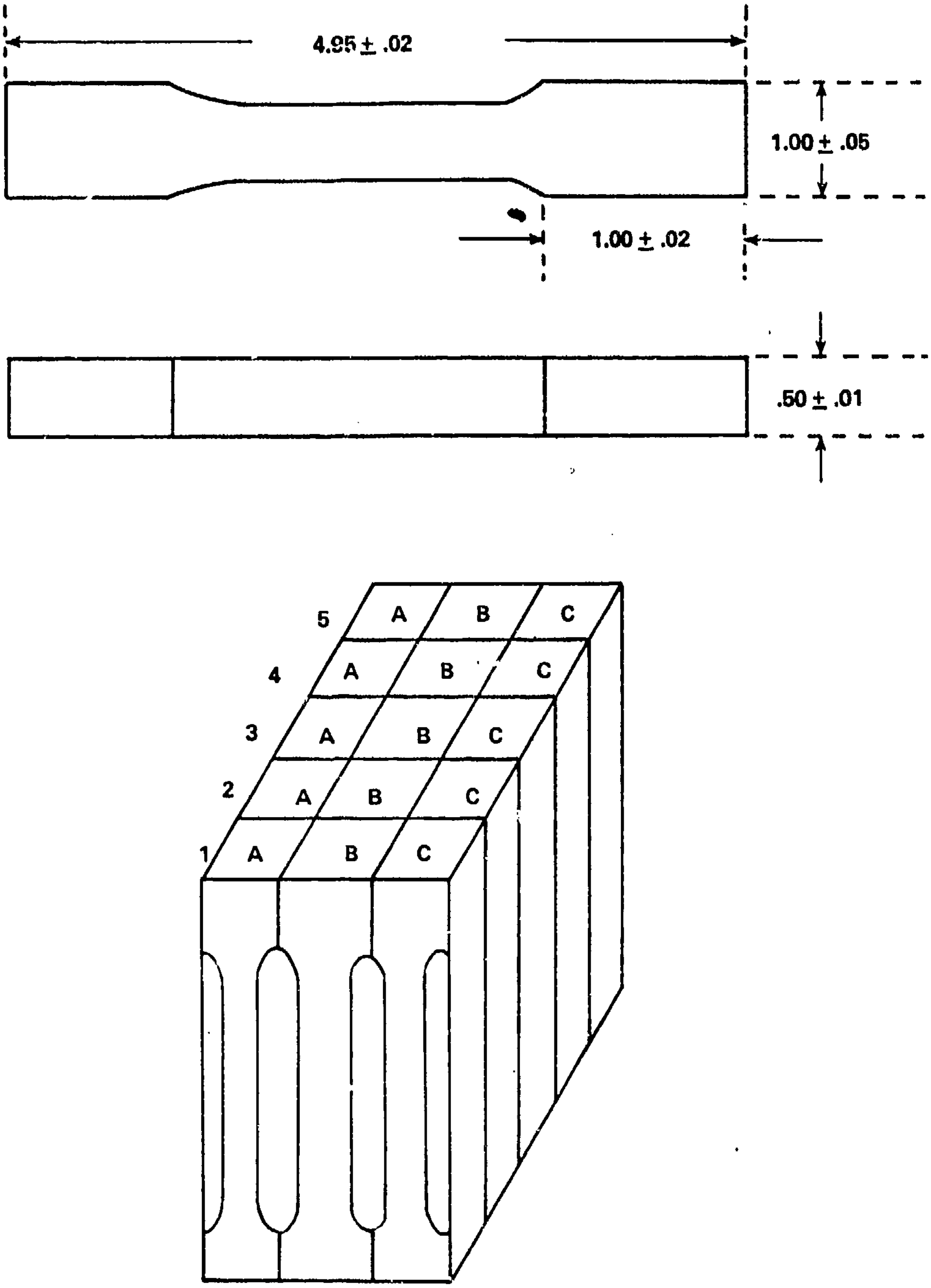


Figure 2. Propellant Block and Individual Tensile Specimen

Manufacturing Company circular saw. Slabs of propellant with a thickness roughly that of the finished dogbone are cut from the appropriate propellant block. This slab is then machined with an Index Machine and Tool Company fly spec milling machine till its thickness conforms to specifications. The final dogbones are then stamped from these slabs and are ready for testing. The dogbones derived from the propellant blocks are numbered and lettered as shown in Figure 2. Testing on the Instron is done by triplicate runs using three dogbones processed from the same slab. For example, the three dogbones numbered 5A, 5B, and 5C are tested at identical conditions. The data resulting from these three dogbones is averaged for presentation.

This process, as described here, sounds very quick and simple. Complications, however, can set in. The very nature of the material being cut and milled make the process hazardous and necessitates remote control of the operation. Such precautions proved necessary when a fire and explosion occurred on 28 March 1976 resulting in severe damage to equipment,but no injury to the personnel operating the equipment. Appendix II contains more information on this incident.

Once the dogbones are formed, they leave the purview of Project NX since mechanical properties testing is the responsibility of Project NE. An engineering request (ER) written by the project engineer of Project NX accompanies the dogbones to Test Area 1-30 for Instron testing. The ER calls out the temperature and cross-head speed of the Instron at which each dogbone will be tested. There are presently two Instron testing devices in operation at Test Area 1-30. Specifications on these devices are given in Figure 3.

To coordinate the cutting and testing of 17 different propellants over a two-year period requires a very large master plan. The basic test philosophy that went into creating this plan is as follows: All of the propellants shall reside in the ovens for the same length of time. That is, the number of days from insertion into the ovens until testing shall be the same for all propellants. Therefore, the basic test plan for all propellants put into the ovens is the same. Since the storage, cutting, and testing facilities could not accomodate all of the propellants at once, the schedule for insertion of the propellant into the ovens had to be staggered. With the 17 propellants Project NX started with, the stagger from the insertion of the first propellant to the seventeenth propellant was over a year. Some idea of the complexity of the master plan can now be seen since the stagger

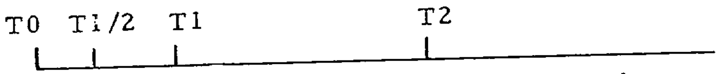
Model No.	Instron TT-C	Instron TM
Full scale load range	2g - 10,000 lb	2g - 200 lb
Chart speed range (in/min)	0.2 - 50.	0.2 - 50.
Testing speed range (in/min)	.02 - 20.	.02 - 50
Temperature range	100°F to 600°F ±2°F	-100°F to 600°F ±2°F
Initial heat-up time (Ambient to 600°F)	40 min	40 min
Initial cool down time (Ambient to -100°F)	30 min	30 min

Figure 3. Specifications of the Instron Test Apparatus

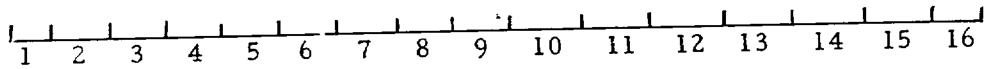
causes different start dates for the formal two-year aging period of each propellant. The stagger could not be made uniform (i.e., start a new propellant each week) since the overlap of two schedules could cause an intolerable number of tests to be performed in one week. A uniform stagger could also cause tests downstream to be performed during Christmas week in order to conform to the basic test philosophy as described above. Each propellant testing schedule had to be treated individually and the earliest possible start date for insertion into the ovens had to be determined. A quick and simple graphical technique for accomplishing this purpose was developed. A test plan for each propellant is slid along a time scale drawn on a long piece of graph paper until an appropriate start time can be determined. Information is then transferred to the graph paper concerning the dates at which propellant will be drawn from the ovens and tested. Figure 4 shows this process in more detail. Any conflicts between propellant schedules can be visualized as well as any test dates on holidays. Thus, the best possible start time for each propellant can be quickly determined.

The generalized test plan that is the same for all propellants placed in the environmental chambers is as shown in Figure 5. The time is the number of months from insertion into the ovens. The different ovens numbered 1 through 6 are set at different operating conditions of constant temperature and relative humidity. Appendix III contains a Standard Cutting Procedure that explains this more fully and presents the test plan in detail as it presently stands. Oven #5 is the stand-by oven that can take over the duties of any one of the other ovens in the event of a malfunction.

As an example, at time point T_6, the propellant has been in the ovens for six months. Samples would be taken from ambient storage and from ovens #3 and #4. They would be tested according to test series S, which is also called the short test series. The engineering requests that would be generated by a propellant being tested at time point T_6 would be as shown in Figure 6 and 7. The first ER directs Test Area 1-21 personnel to extract half-blocks of propellant from the three storage sites involved in T_6 and to cut, mill, and stamp these blocks into dogbones. The second ER directs Test Area 1-30 personnel to test these samples according to the short test series. The day for cutting and the day for testing is called out on the ER's so that coordination between the two groups can be obtained. The days must also be specified so that there is a standardization of the length of time that



Time line with the general test plane written on it



Time line with one one week per division

In practice, the two time lines will have the same amount of time per unit distance. The upper time line is slid along the lower one until a suitable time zero (T0) or start point for a propellant is found. Information is then transferred onto the lower time line as to the time for each test point for that propellant.

Figure 4. Details of the Scheduling Process

Time	Ambient Humidity	90% RH	20% RH	30% RH	50% RH	Ambient Humidity
	77°F		135°F			170°F
T0	F					
T1/2				S	S	
T1		S	S	S	S	S
T3	S	S	S	S	S	S
T6	S			S	S	S
T9		S	S			
T12	F	S	S	S	S	
T24	F	S	S	S		

S - Short Test Series

Instron Test Settings

Sample No.	Temp (Deg. Far.)	X-head Speed (in/min)
1:A, B, C	77	2.0
2:A, B, C	77	2.0
3:A, B, C	165	2.0
4.A, B, C	-65	2.0

Samples 5:A, B, C are to be used as backup spares and not to be otherwise used.

F - Full Test Series

Instron Test Settings

Sample No.	Temp (Deg. Far.)	X-head Speed (in/min)
1,A, B, C	77	2.0
4:A, B, C	165	2.0
5:A, B, C	135	2.0
6:A, B, C	40	2.0
7:A, B, C	0	2.0
8:A, B, C	-40	2.0
9:A, B, C	-65	2.0
10:A, B, C	165	0.2
3:A, B, C	-40	20.0

Samples 2:A, B, C are to be used as backup for above tests. If not needed as spares, test at 77°F and 2 in/min.

Environmental Chamber No.	Temperature Setting	Humidity Setting
1	77°F	90% RH
2	135°F	20% RH
3	135°F	30% RH
4	135°F	50% RH
6	170°F	Ambient (at present)

Chamber No. 5 is held in reserve in the event of failure of one of the other chambers.

Figure 5. General Test Plan

ENGINEERING REQUEST

ENGINEERING REQUEST			DATE
TO:	FROM:	THRU:	
DATE REQUIRED	PROJECT NAME Propellant Aging	PROJECT NUMBER 305908 NX	
SUBJECT JANNAF SAMPLE PREPARATION		DATE COMPLETED	COPIES TO:

ER #________

Propellant Name	Batch Number	Day Cut	Oven #	Block Size	Propellant Name	Time Point
UTX-14420	5/3	Monday	4	Short	H	T_6
UTX-14420	5/2	Monday	3	Short	H	T_6
UTX-14420	5/2	Monday	1	Short	H	T_6
UTX-14420	5/2	Monday	Ambient	Short	H	T_6

Figure 6. Engineering Request for JANNAF Sample Preparation

ENGINEERING REQUEST		DATE
TO:	FROM:	THRU:
DATE REQUIRED	PROJECT NAME Propellant Aging	PROJECT NUMBER 305908 NX
SUBJECT JANNAF SAMPLE TESTING		DATE COMPLETED / COPIES TO:

ER #.

Propellant Name	Batch Number	Day Tested	Oven #	Test Series	Time Point	Sol Gel
UTX-14420	5/3	Mon	4	Short	T_6	
UTX-14420	5/2	Tue	3, 1, and Ambient	Short	T_6	

SHORT TEST SERIES

Sample No.	Temp. (Deg. Far.)	X-head Speed (in/min)
1:A, B, C	77	2.0
2:A, B, C	77	2.0
3:A, B, C	165	2.0
4:A, B, C	-65	2.0

Samples 5:A, B, C are to be used as backup spares and are not to be otherwise used.

FULL TEST SERIES

Sample No.	Temp. (Deg. Far.)	X-head Speed (in/min)
1:A, B, C	77	2.0
4:A, B, C	165	2.0
5:A, B, C	135	2.0
6:A, B, C	40	2.0
7:A, B, C	0	2.0
8:A, B, C	-40	2.0
9:A, B, C	-65	2.0
10:A, B, C	165	0.2
3:A, B, C	-40	20.0

Samples 2:A, B, C are to be used as backup spares for above tests. If not needed as spares, test 77°F and 2 in/min.

Figure 7. Engineering Request for JANNAF Sample Testing

AFRPL FORM 5 OCT 72 REPLACES RPL FORM 0-6, MAR 70, WHICH WILL BE USED

a sample spends between its extraction from the ovens and its testing. Samples stored in oven #4 would lose the moisture gained from storage in the high temperature - high humidity environment of oven #4. The loss of moisture would cause a change in measured mechanical properties. To circumvent this change, propellant from oven #4 is cut in the morning and tested in the afternoon. It is not possible to maintain such a schedule with all of the chambers because of man-power and facility limitations. The cutting/testing schedule that is followed for the ovens at their present settings is as follows for propellants extracted and cut on some day D:

Storage Condition	Days for Testing
ambient	D+1 to D+4
oven #1	D+1
oven #2	D+1
oven #3	D+1
oven #4	D
oven #5	D+1

Another standardization of procedure implemented in this program is what to do in case one of the large blocks (approximate dimensions 5 in. x 8 in. x 3 in.) taken from ambient storage for a full (30 dogbone) test series is not large enough to provide all the dogbones necessary. The simplest procedure is to renumber the dogbones , e.g., designating slab 4 as slab 7 even though it comes between 3 and 5. This procedure simplifies matters for the Instron testing phase. The actual steps are as follows:

No. of Dogbones	Original Slab No.	Redesignated Slab No.
27	2	10
24	6	9

This way the less important parts of the full test series are eliminated.

The example ER shown in Figure 7 will result in 36 dogbones being tested. Each one of these dogbones generates nine numbers upon testing which means that this ER will reduce about 13 lbs of solid propellant 324 numbers. During the two-year formal aging period, a single propellant will cause around 3300 new numbers to come into the world. For the propellant now on hand at Test Area 1-21, around

50,000 numbers will be presented to the project engineer over the three years required for testing all of the propellants. This is around 50 numbers per day, seven days per week, for three years. Keeping track of all these numbers can lead to problems. This problem is compounded by the fact that even though the program has been in progress for a year, the numbers have only recently started coming in. Thus, the project engineer immediately has to contend with approximately 10,000 numbers. Therefore, the methods that will be employed to handle the data are only just beginning to make themselves known. The techniques to be used for cataloging the data will be greatly influenced by the data acquisition system used in conjunction with the Instron. This system has not yet been finalized nor is there any firm date for its completion. Ultimately, a computerized system will have to be employed. Presently, a hard copy computer output is received by the project engineer from the personnel at TETP responsible for data reduction. The output consists almost entirely of numbers with little alphameric information on it. This puts the burden on the project engineer to identify the origin of the numbers as to propellant name, dogbone number, storage condition, and length of time at the storage condition. Project NE, Mechanical Properties Testing, is making progress in this direction. Meanwhile, this information must be extracted from the handwritten data sheets generated at the time the tests are performed. This unfortunate method of gathering is a result of the temporary system presently being used in conjunction with the Instron.

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APPENDIX I

CHALCO ENGINEERING MAINTENANCE RESPONSIBILITIES

Chalco is responsible for performing the following tasks on a periodic maintenance basis for Environmental Chambers #1, #2, #3, and #4.

Chambers #1 and #2 (Tenney Chambers)

Maintenance Requirements	Frequency
Check condition of wicks for proper operation	Weekly
Inspect fan and motor	Monthly
Check for unusual vibration or noise	Monthly
Inspect water system	Monthly
Change wicks	Monthly
Inspect wiring, heaters, insulation, and connections	Monthly
Change water filter	Quarterly
Clean motor fan and housing exterior	Quarterly
Clean heaters and control equipment	Quarterly
Clean water chambers, lines, coils, trays and float valve	Semi-annually

The maintenance requirements for chambers #3 and #4 (Missimer Chambers) are as above except for the deletion of the last requirement to clean the humidification equipment (water chambers, lines, coils, etc.) on a semi-annual basis.

Environmental Chambers #1 and #2, manufactured by Tenney Engineering, have experienced considerable difficulties over the past year. Repairing the chambers required extensive component replacement and modification of the chamber circuitry. Stated below is a history of the repairs made on these two chambers.

Chamber #2

15 April 1975	Controller failed. New controller from Tenney received defective. Reordered replacement.
23 April 1975	Replaced recorder

15 May 1975	Replaced controller
17 June 1975	Controller failed
20 June 1975	Replaced controller
7 October 1975	Replaced sensor and controller
8 October 1975	Replaced solenoid valve (SV-1)
7 November 1975	Replaced compressor and expansion valve
12 November 1975	Installed service valves for refrigeration system
24 February 1976	Completed modification to control and refrigeration system consisting of:
	Replaced 1 each solenoid valve with 2 each solenoid valves (SV-4 and SV-11)
	Replaced 2 each capillary tube assemblies with filter driers
	Replaced 1 each constant pressure valve
	Installed 30 amp line circuit breakers
	Installed 2 each heater relays
	Installed 1 each 1 Amp circuit breakers
	Installed 2 each 15 Amp circuit breakers
	Installed 230 VAC transformer
Chamber #1	
29 October 1975	Replaced controller

APPENDIX II

ACCIDENT AT TEST AREA 1-21

1. On 28 Mar 74 at 1045 hours, a fire and explosion occurred in Bldg 8582, Room B, Solid Propellant Milling and Cutting Facility.

2. Two operators were present; MSgt Louis A. Franks and Mr. Kelley N. Palmer. No injuries occurred.

3. The incident occurred while remotely cutting an eight pound piece of TP-H8219 propellant (about 9" X 7" X 4") into one-half inch slabs with a band saw. MSgt Franks noticed a flame near the band saw table in the region of the saw blade. After he had stepped back from the protective window, an explosion occurred. The TP-H8219 specimens being cut at the AFRPL were relatively new samples that were entering an aging program. This propellant contains ultrafine ammonium perchlorate (20%), iron oxide (1%), 90 micron ammonium perchlorate and 6 micron ammonium perchlorate. A Class II explosive classification resulted from AFRPL test on compositions similar to TP-H8219.

4. An AFRPL group closely inspected the site on 2 Apr 74. Physical evidence showed that the block of propellant being cut burned without detonation. The conflagration caused substantial but repairable damage to the band saw. A cutting exhaust duct,4 inches I.D. with 0.125 inch walls connected to the band saw with a two-inch flexible line, exploded scattering large metal fragments throughout the cell. Another more or less simultaneous explosion occurred under a vacuum holding plate on an adjacent milling machine blowing the vacuum plate against the ceiling. The milling machine was also in repairable condition after the incident. The cutting cell door was blown off and the door of an adjacent cell pulled off, with damage to lower hinges.

5. Damage was sustained by the band saw, milling machine, exhaust duct, a small vacuum pump, two doors and electrical wiring. Estimated cost of damage was:

a.	Band saw	$ 280.00
b.	Milling machine	300.00
c.	Exhaust duct	700.00
d.	Vacuum pump	100.00
e.	Doors	350.00
f.	Electrical & cleanup	3,410.00
	TOTAL	$5,140.00

6. The incident was probably initiated with a fire caused by friction between the saw blade and the propellant. An examination of a piece of propellant that had been previously cut off the block that was involved in the incident showed that extensive deformation and smearing of the aluminum particles had occurred. Thus, galling between the large aluminum particles (90 micron) and the saw blade could have created sufficient local heating to cause ignition. Burning particles from

the burning propellant were swept into the cutting exhaust duct igniting an accumulation of finely divided propellant in an approximately ten-foot horizontal duct section. The propellant particles in the duct exploded. By an undetermined process, a spark also ignited finely divided propellant powder accumulated in a cavity under a vacuum holding plate on the milling machine bed. The ensuing explosion ripped the vacuum holding plate off the machine.

7. Several recommendations were made by the investigators for propellant cutting operations in the future. These recommendations were primarily involved with prevention of propellant particle accumulations and ease of cleaning and inspection of the equipment.

a. Replace the band saw with a large circle saw. This was suggested because the band saw is a more complex device that is difficult to clean and has a large number of places where entrained propellant particles can be subjected to pinching and friction. Particularly, particle entrapment between the driving wheels and the saw blade appeared to be an ignition hazard.

b. Use a tungsten carbide tipped saw blade. Galling between large aluminum particles (90 micron) in the propellant and a slightly dulled saw blade appeared a probable ignition source for this incident. Use of tungsten carbide cutters, which are not readily dulled from cutting aluminum, could reduce the amount of local heating during cutting. Cutting speed did not stand out as a factor in this incident but should be controlled to less than 1000 feet per minute.

c. Design the particle exhaust system so that the inlet at the cutter is not reduced to less than fifty percent of area of the primary exhaust duct. This design feature will keep the air velocity and mass flow in the exhaust duct maximized. Such conditions should aid in keeping particles from accumulating in the duct. The system before this incident used a four inch I. D. duct terminating at the cutter with a two inch I. D. flexible hose.

d. Minimize horizontal exhaust duct sections to less than two feet and eliminate dead air spaces in connections where particulate material could accumulate. An explosion occurred in a long horizontal duct section where propellant dust from the cutting operation had settled out. If the exhaust duct was constructed so that it rose at a steep angle to a high point and then fell at a sharp angle to the dust collector, buildup of particulate in the duct would be lessened. Also, the duct in use has horizontal connections for accessory equipment with dead air spaces about one foot long at right angles to the duct flow between the duct and shut off valves. Such dead air zones that would be efficient particle collectors should be eliminated.

e. Flush cutting exhaust duct with high pressure nitrogen each day following cutting operations. By flushing high pressure nitrogen through small lines installed in the rising portion of the exhaust duct after each day of cutting operation, dangerous buildup of fine propellant particles would be avoided. Direct observation of the duct interior for inspection would be desirable.

f. Use lightweight conductive material for construction of the exhaust duct. The duct in use was constructed of one-eighth inch thick stainless steel with heavy flanges at duct connections. This mass of metal contributed to the physical damage

in the cell when the duct exploded. Lightweight construction should not impair operation of the duct and would lessen damage to surrounding equipment due to impact of heavy metal fragments.

g. Construct vacuum plate on milling machine with attached hinges so that the plate can be readily lifted for cleaning vacuum cavity under plate. The vacuum holding plate on the milling machine had many bolts to keep it in place and maintain a vacuum seal. Accumulated propellant material under the plate made possible the explosion that lifted off the plate. By use of an inset O-ring gasket and positioning pins below the vacuum plate, bolting would not be necessary as the vacuum pressure would hold the plate down. By lifting the plate, easy access to the vacuum cavity for clean up would be obtained. Since dropping the plate could be hazardous to an operator, use of a hinged mechanism on the plate would reduce that possible danger.

h. Remove stored propellant from adjacent cell. Four hundred pounds of Class II propellant was stored in the cell adjacent to the chamber where the explosive incident occurred. The lower hinges of the storage cell door were damaged and the door was forced open. Although the contents of the cell were undisturbed, a large fire could have occurred had the stored propellant become involved.

i. An Air Force Rocket Propulsion Laboratory representative should be sent to industrial facilities for assessing the relative merit of their propellant cutting operations versus that of the AFRPL. Thiokol at Huntsville, Alabama has not had a fire during propellant cutting in the last ten years. Thiokol's facility should be a good candidate for obtaining improved cutting procedures.

APPENDIX III

GENERAL AND DETAILED TEST SCHEDULES

Time Point	Propellant Type	
T_o	H	Cut one whole block of propellant taken from ambient storage and mark as usual. Place half-blocks in Environmental Chambers as follows:

Chamber	No. of Half-Blocks
#1 77°F/90% RH	5
#2 135°F/20% RH	5
#3 135°F/30% RH	6
#4 135°F/50% RH	5
#6 170°F/Ambient	3

T_o	A	Cut and mark one full block from ambient storage. No blocks are to be put in Environmental Chambers.
$T_{1/2}$	H	Remove one half-block from Environmental Chambers 3 and 4. Cut and mark as usual.
T_1	H	Remove one half-block from Environmental Chambers 1, 2, 3, 4, and 6. Cut and mark as usual.
T_3	H	Remove one half-block from Environmental Chambers 1, 2, 3, 4, and 6. Cut and mark as usual. Remove one half-block from ambient storage. Cut and mark as usual.
T_3	A	Remove one half-block from ambient storage. Cut and mark as usual.
T_6	H	Remove one half-block from Environmental Chambers 3, 4, and 6 and one half-block from ambient storage. Cut and mark as usual.
T_6	A	Remove one half-block from ambient storage. Cut and mark as usual.
T_9	H	Remove one half-block from Environmental Chambers 1 and 2. Cut and mark as usual.

Time Point	Propellant Type	
T_{12}	H	Remove one whole block from ambient storage and one half-block from Environmental Chambers 1, 2, 3, and 4. Cut and mark as usual.
T_{12}	A	Remove one whole block from ambient storage. Cut and mark as usual.
T_{24}	H	Remove one whole block from ambient storage and one half-block from Environmental Chambers 1, 2, and 3. Cut and mark as usual.
T_{24}	A	Remove one whole block from ambient storage. Cut and mark as usual.

Type H propellant is stored in the Environmental Chambers as well as under conditions of ambient temperature and humidity.

Type A propellant is stored only under conditions of ambient temperature and humidity.

PROPELLANT AGING - DETAILED TESTING SCHEDULE FOR FY-75 & FY-76								
TIME	T_0	$T_{1/2}$	T_1	T_3	T_6	T_9	T_{12}	
Propellant								Type
UTP-3001	3 Feb 75	17 Feb 75	3 Mar 75	28 Apr 75	21 Jul 75	13 Oct 75	5 Jan 76	H
TP-H8221	24 Feb 75	-	-	19 May 75	11 Aug 75	-	26 Jan 76	A
UTX-14420	10 Mar 75	24 Mar 75	7 Apr 75	2 Jun 75	25 Aug 75	17 Nov 75	9 Feb 76	H
UTX-14422	24 Mar 75	7 Apr 75	21 Apr 75	16 Apr 75	8 Sep 75	1 Dec 75	23 Feb 76	H
UTX-14424	12 May 75	26 May 75	9 Jun 75	4 Aug 75	27 Oct 75	19 Jan 76	12 Apr 76	H
ANB-3432	26 May 75	-	-	18 Aug 75	10 Nov 75	-	26 Apr 76	A
TP-H8220	9 Jun 75	23 Jun 75	7 Jul 75	1 Sep 75	24 Nov 75	16 Feb 76	10 May 76	H
ANB-3434	11 Aug 75	-	-	3 Nov 75	26 Jan 76	-		A
TP-H8219	8 Sep 75	-	-	1 Dec 75	23 Feb 76	-		A
TP-H1135	20 Oct 75	3 Nov 75	17 Nov 75	12 Jan 76	5 Apr 76	28 Jun 76		H
TP-H8216	2 Feb 76	-	-	26 Jan 76				A
ANB-3428	1 Mar 76	15 Mar 76	29 Mar 76	24 May 76				H
TP-H8213	8 Mar 76	22 Mar 76	5 Apr 76	31 May 76				H
TP-H8214	7 Jun 76	21 Jun 76						H

AFSC FORM JUL 61 185b GENERAL PURPOSE WORKSHEET (10½" X 8") PREVIOUS EDITIONS OF THIS FORM ARE OBSOLETE.

APPENDIX IV

PROPELLANT INGREDIENTS BEING AGED

POLYMERS

HTPB	hydroxy terminated polybutadiene
CTPB	carboxy terminated polybutadiene
PBAN	polybutadiene - acrylic acid - acrylonitrile terpolymer
R-45M	a free radical produced HTPB
Butarez-HTS	a lithium initiated HTPB with secondary hydroxy groups.

CURING AGENTS

DDI	dimer acid diisocyanate
HDI	hexamethylene diisocyanate
IPDI	isophorone diisocyanate
TDI	toluene diisocyanate

PLASTICIZERS

DOA	dioctyl adipate
IDP	isodecyl pelargonate
Oronite	a polybutene
Stan-Pete	a hydrocarbon oil

CURE CATALYSTS/SUPPRESSORS

DBTDL	dibutyl tin dilaurate
BA	benzilic acid
$Cr\ Cl_3$	chromium trichloride
ZnO	zinc oxide
LA	linoleic acid
$Fe\ (AA)_3$	ferric acetyl acetonate

BONDING AGENTS

HX-752	propylene imine adduct of isophthalic acid
TEPAN	reaction product of tetraethylene pentamine and acrylonitrile
TEPANOL	reaction product of TEPAN and glycidol

STABILIZERS

DTBH	ditertiarybutyl hydroquinone
Agerite White	di-beta-naphthyl-p-phenylenediamine
PBNA	phenyl-beta-naphthylamine
Plastinox 711	di(tridecyl) thiodipropionate
A02246	2-2'-methylene-bis(4-methyl-6-t-butyl phenol)
Pro-Tech 6402	a Chemical System Division (UTC) ingredient
Pro-Tech 3102	a Chemical System Division (UTC) ingredient
S	sulfur
UOP-36	N-phenyl-N'-cyclohexyl-p-phenylene diamine

OXIDIZERS

AP	ammonium perchlorate
HMX	cyclotetramethylenetetranitramine

FUEL

Al	aluminum